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A sampling device, for obtaining samples of highly radioactive uranyl sulfate solution, was developed for use in the HRT. Conceptual design was by C. L. Segaser of REED Design Section; detailed design by R. Rosenberg, C. L. Terry and T. L. Trent of the ORNL Engineering Department. Assisting in the test work was W. B. Krick.

A prototype sampler, incorporating virtually all the modifications indicated by earlier test work, was built and successfully operated in conjunction with a low pressure water system and a high pressure, high temperature uranyl sulfate system.

The major portion of the development work was applied to the chamber which isolates the sample and to the receptacle which receives the sample from the chamber for delivery to the analytical facilities. Tests made with an early prototype of the isolation chamber formed the basis for modifications to it which resulted in 1) a sample volume consistent with available shielding and 2) more nearly representative samples of the small amount of solids carried by the sampled stream. Performance of the early model of the receptacle (flask and holder) indicated changes necessary to provide a vacuum-tight seal between receptacle and isolation chamber and minimize deposition of sample drops on the flask.

MECHANICAL DESCRIPTION

Samples of liquid and suspended solids are taken from the high and low pressure systems of the HRT. Locations of the four sampling points are shown on the system flowsheet¹. When a sample is taken from the high pressure system, liquid flows from high pressure through the samplers to the low pressure dump tanks. Samples are taken from the low pressure system by pressurizing the dump tanks and forcing liquid through the samplers into a fuel transfer tank.

One heavily shielded sample station of the type shown in Figure 1 is provided for each of the reactor systems - core and blanket. Each station contains two isolation chambers: one for isolating samples from the low-pressure system and the other for obtaining samples from the high pressure system. Each

chamber in the station is served by a common loading and manipulating device.

The fluid being sampled flows through the sampler when the inlet valve is opened, and passes through the isolation chamber to the low pressure system. When the sampler has been flushed sufficiently to remove traces of the previous sample, the inlet valve is closed and the vent valve is opened, thereby permitting the fluid to drain to the overflow level of the isolation chamber. The trapped sample remains in the isolation chamber for about 15 minutes. Meanwhile, the holder containing the evacuated flask is lowered through the loading tube to the transfer mechanism. The assembly is then indexed under the proper isolation chamber where the flask holder is raised by an air cylinder until contact is made between the isolation chamber nozzle and the inverse cone on the carrier head (Figure 2). Further travel of the flask holder causes the hypodermic needle to puncture the rubber diaphragm and seats the carrier head against the nozzle. The sample is discharged into the flask by opening the valve on the isolation chamber. When the sample is in the flask, the procedures are reversed and the flask holder is removed into a shielded carrier for transport to the Analytical Laboratory.

EARLY DEVELOPMENT WORK

Isolation Chamber

Since shielding available at the hot laboratory dictated a sample size of not more than 5 mls, it was necessary to eliminate a hand valve immediately downstream from the isolation chamber since this valve would result in a total sample volume of approximately 12 mls. The inlet hand valve and isolation chamber were shown in early drawings connected to the vent valve by an inverted "U" tube. This arrangement was mocked-up with 1/8-in. I.D. glass tubing connected to a prototype isolation chamber to study the venting action. It was found that this arrangement resulted in trapping a gas-liquid mixture in the leg which prevented the isolation chamber from draining to its overflow level. The vent valve was raised above the isolation chamber elevation and connected to it by a continuously sloping vent line as suggested by J. A. Watts. The arrangement resulted in proper

drainage and a sample size of 4.5 to 5.0 ml.

The fuel solution will contain fission product solids of the order of 0.1 per cent by weight. In order to determine the quality of sample taken by the HRT sampler under these conditions, a slurry of ThO_2 , 0.1 per cent by weight, was circulated through the sampler as shown schematically in Figure.3. A prototype isolation chamber was tested. Solution was circulated through the chamber at 0.5 gpm and full stream samples were taken at points A and B prior to closing the valve. The sample was retained in the isolation chamber for 15 minutes before being withdrawn into the flask. This retention time at temperature is dictated by the relative insolubility of uranium peroxide at higher beta power density². Table 1 illustrates the deviation of the sample analyses from the stream analysis. Comparison of results at 3 minutes retention time and 15 minutes retention time demonstrates that increased accuracy in solids sampling accrues from shorter retention time; therefore, this interval should be as short as possible, consistent with the limitations imposed by the beta power level.

The most accurate sample of fission product solids will be obtained by withdrawing the sample immediately after the sample is isolated, and dump tank pressure reached. Gas evolved in the sample flask can be vented at the degassing screw before raising the flask holder into the carrier.

The following revisions were made to the isolation chamber in an effort to improve its ability to deliver a more nearly representative sample of solids:

- 1) Scallops were milled in the stem stop of the isolation chamber in order to promote more rapid drainage to the overflow level after closing the inlet valve.
- 2) Two vent holes were drilled in the sleeve of the isolation chamber to induce more rapid removal of the sample into the flask. The holes vent the outer annulus of the chamber.
- 3) Disassembly and inspection of the isolation chamber after runs made with 0.1 per cent ThO_2 slurry had repeatedly shown ThO_2 collected on the small land in the bottom of the chamber. This was finished to a smooth contour to allow

the solution to sweep the settled solids into the sample flask upon opening the chamber nozzle. Table II indicates that solids sampling inaccuracy was hereby reduced to about 30 per cent.

Receptacle

The first problem to be overcome in debugging the flask and flask holder assembly was maintaining a vacuum in the flask until the holder is brought into contact with the isolation chamber nozzle and the chamber opened. Unless a vacuum of half-atmosphere or greater is present, the full volume of isolated sample will not enter the flask.

The elastomer to be used for the flask diaphragm must be resilient enough to seal the puncture made by the evacuating needle. Since radiation from the flask holder might require its assembly and evacuation in a shielded area, it was desirable to employ a flask closure which would be vacuum tight for a period of hours. Accordingly, a number of diaphragm materials were tested for vacuum retention using a flask and cap. Gum rubber diaphragms which had been punctured by 18 gauge evacuating needles leaked virtually no air over a 24-hour period. Other diaphragm materials tested, including Buna-N rubber and several silicone rubbers, were notably inferior in this respect. Pure gum rubber diaphragms 1/8-in. thick were determined to be most satisfactory from the standpoint of vacuum retention and ease of puncture.

The stainless-to-stainless seal between isolation chamber nozzle and plunger, Figure 2, was found to require far too great a sealing force to be practical. The test criterion was that the flask pressure remain below 0.5 atm. for 15 minutes with the plunger engaged with the chamber nozzle. Both polyethylene and Teflon gaskets were tested and found to work almost equally well; with either material the test conditions are met. In order to insure the minimum sealing force of 50 pounds, the design of the plunger spring was altered to provide a pre-loading of 60 pounds.

Throughout the course of this test work, the deposition of one or several drops of sample solution on the flask diaphragm was a frequent occurrence.

Presumably, this resulted from solution draining down the chamber wall into the needle after the sample was drawn into the flask. A larger diameter hypodermic needle, No. 12 gauge, was tried in an unsuccessful attempt to correct this. Metal supports for the rubber diaphragm were tried, and the speed of cylinder travel was varied, with no improvement. Finally, vent holes were drilled in the plunger and body of the flask holder in order to prevent the drop of solution being pulled out of the needle when the plunger rises in the holder.

PROTOTYPE SAMPLER ASSEMBLY TEST

A prototype sampler plug assembly, incorporating all changes suggested by work discussed above, was fabricated to prove the operation of the sampler as a complete device. This prototype was installed in a test stand at building 9204-1 and connected to a low pressure water circulating system. When the sampler was demonstrated to take samples satisfactorily from this system, the prototype was connected to the HRT mockup with appropriate piping, as indicated in Figure 4. A total of more than 170 samples of 10 g U/l uranyl sulfate were taken from the high pressure, high temperature region of the HRT mockup. Sample volume rarely varied from the 4.5 - 5.0 ml range. Only about 10 per cent of the sample flasks removed from the sampler had as much as one drop of solution on the flask diaphragm.

Operation of the sampler was satisfactory except for the following:

- 1) Flow control valve HCV-136 required throttling by adjustment of operator air pressure. When wide open, the sampler letdown exceeded the capacity of the mockup feed pumps. This was not a problem of sampler development, as such, but does indicate that attention should be given to obtaining more reliable flow control.

- 2) Infrequently, no sample fluid was driven into the flask. The reason for failure to obtain a sample on occasion, where operator error was not the cause, was ascribed to failure of the flask holder to index with the isolation chamber. Indexing difficulties had been noted from time to time, and were ascribed in every case to insufficient rigidity in the indexing rod and mechanical linkages.

This shortcoming had been partially remedied by tacking the universal joints with weld metal to remove play. The design for the indexing mechanism was changed to the extent of increasing the indexing rod diameter and eliminating the universal joints.

Several miscellaneous pins and fittings were not rugged enough for dependable service; these fittings were marked for revision on design drawings.

It was apparent when the prototype plug was connected to the piping at the test stand that it would be virtually impossible to make the remote connections with ordinary back-up rods. Therefore, adjustable guide brackets for the rods were located on the plug underside, and a double universal joint linkage inserted in each rod.

HRT Bellows-Type Hand Valves and Isolation Chamber

The inlet valve and vent valve at each sampling point are bellows-type block valves manufactured by the Fulton-Sylphon Division of Robertshaw-Fulton Controls Co. to their Drawing 10895R. These valves are designed for 2000 psi service and feature an integral .076-in. seat and Stellite No. 6 plug. They are intended primarily for shut-off service.

The isolation chamber (ORNL Dwg. D-21577) is an adaptation of the design proposed by C. L. Segaser³. It also has a Stellite-tipped plug.

Both the inlet valve and the isolation chamber on the prototype sampler were opened and closed the order of 600 cycles during the course of this work.

A hydrostatic test of the isolation chamber was made after approximately 300 cycles, including about 80 cycles on 80 - 90°C uranyl sulfate solution. At 2000 psi no leakage was observed from the nozzle over a 10 minute period, with 80 inch-pounds torque applied to the stem. A final hydrostatic test resulted in a leakage of 0.25 - 0.50 drops/minute under the same conditions.

At the conclusion of the test work, the inlet valve leaked ~0.25 drops/minute at 2000 psi differential with 120 inch-pounds applied to the stem. Friction between the shaft and the shield plug penetration was noticeably greater than in the case of the isolation chamber. In terms of torque actually trans-

mitted to the sealing plug, therefore, the inlet valve probably seals as well or better than the isolation chamber.

Memory Tests

In order to use the samplers for alternately sampling the condensate supply and the fuel, it was necessary to determine how much condensate flush would be necessary to remove traces of uranium from the system before isolating a condensate sample for uranium analysis. The test arrangement is shown in Figure 5. Test procedure is outlined in the Appendix.

Potassium nitrate was the solute employed in this test, since potassium is readily analyzed down to 1 ppm by flame photometry. Figures 6 and 7 indicate that potassium concentration dropped to the range of 1 - 2 ppm after flushing with from 50 to 60 liters of water. The quantity of flush water required was virtually independent of initial potassium concentration, C_i , in the range 3200 - 8400 ppm. Whether the vent valve was open or closed during flushing had no pronounced effect on flushing efficiency; compare Figures 6 and 7. The corresponding uranium concentrations would be $\frac{\text{mol wt. U}}{\text{mol wt. K}} = 6.1$ times the potassium concentrations plotted here.

No means for flushing the outside of the isolation chamber nozzle is provided in this sampler. Therefore, a standard procedure for taking condensate samples should include at least one sample withdrawal prior to withdrawing a sample for analysis. This will serve to remove traces of uranium from the outlet nozzle.

Safety Devices

A degassing screw is provided in order to vent pressure from the flask before removing from the station. A safety-guard is located at the bottom of the loading tube in order to prevent accidentally dropping the flask holder into the sampler well, should the receptacle not be indexed under the loading tube. This guard did not perform satisfactorily on the prototype; it failed to return to the rest position consistently. This trouble has been overcome in the fabrication of the guards for the HRT samplers.

Experience with the prototype sampler indicated that the flask holder receptacle was not always indexed accurately under the isolation chamber with the result that sample solution would be discharged upon the outside of the flask holder. Even though the modifications to the indexing mechanism should prevent system error, operator error is still a possibility. To minimize the possibility of either an electrical switch was mounted in such a way that it is actuated when the flask holder is properly engaged with the isolation chamber nozzle (ORNL Dwg. D-21607). The isolation chamber is opened and closed by a manually operated, non-rising stem. To lessen the possibility of accidentally dumping reactor fluid through the isolation chamber, an electrical switch was attached to the chamber which indicates whether the chamber is open or closed (ORNL Dwg. D-21606).

CONCLUSIONS

The HRT sampler meets the requirements as set forth in the HRT Design Request⁴ except for solids sampling. Samples taken according to the standard sampling procedure are about 30 per cent low in solids compared with the true concentration.

It was impractical to mockup all the piping and equipment associated with the sampler station, but external to it. Therefore, it is possible that when the sampler is tested in the reactor system, some revision in external piping or in operating procedures may be required for satisfactory performance of the sampler.

REFERENCES

1. R. B. Briggs, et al., HRP Quar. Prog. Rep. April 30, 1955, ORNL 1895, p. 5-7.
2. P. M. Wood, "Possibility of Peroxide Precipitation in HRT Dump and Sampler Lines," ORNL-CF-54-11-172, Nov. 29, 1954
3. R. B. Briggs, W. R. Gall, C. L. Segaser, "HRT Design Request No. 5 - High Pressure and Low Pressure Sampling Systems for the HRT," ORNL-CF-54-9-223, Sept. 29, 1954.
4. R. B. Briggs, et al., ibid.

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Attachments

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TABLE I

Sampling Results With 0.1% ThO₂ Slurry,
Prior to Modifying the Isolation Chamber

<u>Run Number</u>	<u>True Conc.,* % ThO₂</u>	<u>Sample Conc., % ThO₂</u>	<u>Absolute Error % ThO₂</u>	<u>Percentage Error</u>	<u>Retention Time, Minutes</u>
12	.110	.110	0	0	3
13	.113	.102	-.011	-9.7	3
14	.110	.102	-.008	-7.3	3
Average percentage error = -5.6% for 3 minutes retention					
19	.073	.043	-.030	-41	15
20	.088	.055	-.033	-37.5	15
21	.086	.049	-.037	-43	15
22	.086	.048	-.038	-44	15
23	.084	.057	-.027	-32	15
25	.094	.049	-.045	-48	15
26	.077	.042	-.035	-45.5	15
28	.084	.033	-.051	-60.6	15
30	.089	.066	-.023	-25.9	15
31	.089	.050	-.039	-44	15
32	.087	.047	-.040	-46	15
33	.080	.048	-.032	-40	15
34	.080	.063	-.017	-21	15

Average percentage error = -40.6% for 15 minutes retention

*Average of analyses made on samples taken upstream and downstream of the isolation chamber immediately prior to isolating a sample.

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TABLE II

Sampling Results With 0.1% ThO₂ Slurry,
Modified Isolation Chamber

<u>Run Number</u>	<u>True Conc.,* % ThO₂</u>	<u>Sample Conc., % ThO₂</u>	<u>Absolute Error % ThO₂</u>	<u>Percentage Error</u>	<u>Retention Time, Minutes</u>
35	.074	.049	.025	-34	15
36	.067	.057	.010	-15	15
37	.067	.056	.011	-16	15
38	.075	.052	.023	-31	15
39	.068	.035	.033	-49	15
40	.069	.052	.017	-25	15

Average percentage error = -28.8%.

*Average of analyses made on samples taken upstream and downstream of the isolation chamber immediately prior to isolating a sample.

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APPENDIXSampling Procedure for Memory Tests

I. "Contamination" Cycle

- 1) Circulate a KNO_3 solution having a nominal potassium concentration of 10,000 ppm through valve No. 1, the sampler, and return through valve No. 2 to the supply tank. Sampler vent valve open or closed; flow rate: 0.5 gpm.
- 2) Take full-stream sample below valve No. 2, then shut off pump.
- 3) Drain KNO_3 solution out of line to sampler. Close valve No. 1
- 4) Open vent valve (if closed) and allow 10 minutes for sample to drain to isolation chamber overflow level.
- 5) Withdraw sample into evacuated flask.

II. Water Flush Cycle

- 1) Close valve No. 2; open valve No. 4.
- 2) Check to see that valve B is closed.
- 3) Fill constant head tank with building water and regulate air overpressure to 10 psi.
- 4) Using valve No. 3 as a throttling valve to limit initial water flow rate to 0.5 gpm, open valve A. Circulate water through the sampler into the hold-up tank until flow ceases, as indicated by the rotameter.
- 5) Close valve A.
- 6) Open valve B and allow hold-up tank to drain.
- 7) Open sampler vent valve (if closed) and allow 10 minutes for the isolation chamber to drain to overflow level. Close vent valve.
- 8) Withdraw sample into evacuated flask.
- 9) Repeat steps 2 through 8 a minimum of five times.

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Fig. 1.

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Fig. 2.

FIGURE 3. Test Arrangement for ThO₂ "Slurry" Sampling.

FIGURE 4. Piping Diagram for Sampling the High-Pressure System of HRT Mockup.

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FIGURE 5. Isometric of Piping for the Memory Tests.

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FIGURE 6. Potassium Concentration vs. Number of Flushing Cycle; Vent Valve Open

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FIGURE 7. Potassium Concentration vs. Number of Flushing Cycle; Vent Valve Closed